



# ITO-MgF<sub>2</sub> Film Development for PowerSphere Polymer Surface Protection

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Multi-kilogram class microsattellites with a PowerSphere electric power system are attractive for fulfilling a variety of potential NASA missions. However, PowerSphere polymer surfaces must be coated with a film that has suitable electrical sheet resistivity for electrostatic discharge control, be resistant to atomic oxygen attack, be transparent to ultraviolet light for composite structure curing and resist ultraviolet light induced darkening for efficient photovoltaic cell operation. In addition, the film must be tolerant of polymer layer folding associated with launch stowage of PowerSphere inflatable structures. An excellent film material candidate to meet these requirements is co-sputtered, indium oxide (In<sub>2</sub>O<sub>3</sub>) – tin oxide (SnO<sub>2</sub>), known as “ITO”, and magnesium fluoride (MgF<sub>2</sub>). While basic ITO-MgF<sub>2</sub> film properties have been the subject of research over the last decade, further research is required in the areas of film durability for space-inflatable applications and precise film property control for large scale commercial production. In this paper, the authors present film durability results for a folded polymer substrate and film resistance to vacuum UV darkening. The authors discuss methods and results in the area of film sheet resistivity measurement and active control, particularly dual-channel, plasma emission line measurement of ITO and MgF<sub>2</sub> plasma sources. ITO-MgF<sub>2</sub> film polymer coupon preparation is described as well as film deposition equipment, procedures and film characterization. Durability testing methods are also described. The pre- and post-test condition of the films is assessed microscopically and electrically. Results show that a ~500Å ITO-18vol% MgF<sub>2</sub> film is a promising candidate to protect PowerSphere polymer surfaces for Earth orbit missions. Preliminary data also indicate that in situ film measurement methods are promising for active film resistivity control in future large scale production. Future film research plans are also discussed.

## Nomenclature

Å	angstrom, 1.0 <sup>-10</sup> m
AO	atomic oxygen
cm	centimeter, 1.0 <sup>-2</sup> m
EDU	engineering development unit
ESD	electrostatic discharge

GRC	NASA Glenn Research Center
ITO-MgF <sub>2</sub>	indium oxide (In <sub>2</sub> O <sub>3</sub> ), tin oxide (SnO <sub>2</sub> ) - magnesium fluoride (MgF <sub>2</sub> )
m	meter
MISSE-5	Materials International Space Station Experiment number 5
mm	millimeter, 1.0 <sup>-3</sup> m
nm	nanometer, 1.0 <sup>-9</sup> m
N	Newton
PEM	plasma emission monitoring
PSA	pressure sensitive adhesive
QCM	quartz crystal monitor
Vol%	volume percent
VUV	vacuum ultraviolet light

## I. Introduction

Multi-kilogram class microsattellites (or microsats) are attractive for fulfilling a variety of potential NASA missions. However, small microsats tend to be power poor due to lack of available surface area on which to mount photovoltaic cells. An excellent design solution for this power generation challenge is the PowerSphere concept<sup>1</sup> shown in Figure 1. The PowerSphere space-inflatable, geodetic solar array provides attitude-independent microsat power with very low mass and efficient launch packaging. The leading PowerSphere inflatable structure technology consists of an ultraviolet (UV) activated resin impregnated fiberglass constrained by a thin polymeric layer. The PowerSphere solar panels are comprised of thin-film photovoltaic cells on polymer substrate, encapsulated with a polymer superstrate and connected with fiberglass hinges. The solar panels are connected to rigid end panels deployed out from spacecraft by inflatable center columns. The column structure is comprised of a fiberglass isogrid impregnated with UV curable resin and is sandwiched between polymer bladder and restraint layers<sup>2</sup>. An Engineering Development Unit (EDU) PowerSphere was recently built, deployed and rigidized<sup>3</sup>. Figure 2 is a photograph of the PowerSphere EDU interior view showing the center column, rigid end plate and solar panels for one hemisphere.

During operation in Earth orbit, virgin polymer surfaces could charge to high voltage leading to damaging electrostatic discharge (ESD)<sup>4</sup>. In addition, the presence of monatomic oxygen (AO) and UV radiation leads to aggressive attack and/or darkening of unprotected polymers. To ameliorate these space environmental effects, PowerSphere polymer surfaces must be coated with a thin protective film. This film must have suitable electrical sheet resistivity for ESD control, be resistant to AO attack, be transparent to UV for composite structure curing and resist UV-darkening for efficient photovoltaic cell operation. In addition, the film must be tolerant of polymer layer folding associated with launch stowage of PowerSphere inflatable structures such as the center columns. An excellent film material candidate to meet these requirements is co-sputtered, indium oxide (In<sub>2</sub>O<sub>3</sub>) – tin oxide (SnO<sub>2</sub>) [a.k.a., ITO] and magnesium fluoride (MgF<sub>2</sub>)<sup>5</sup>. Film characteristics can be tailored by the selected volume percentage (vol%) of MgF<sub>2</sub> (typically 0-30%). Basic ITO-MgF<sub>2</sub> film properties have been the subject of research over the last decade. Yet, further research is required in the areas of film durability for space-inflatable applications and precise film property control for large scale commercial production. To address this need, film development work is in progress<sup>6-8</sup>. In this paper, the authors discuss further film development efforts in the areas of film durability, film sheet resistivity measurement and active control for commercial production.



Figure 1: PowerSphere Concept.

## II. Film Durability Testing

### A. Folded Polymer Substrate

#### 1. Test Article

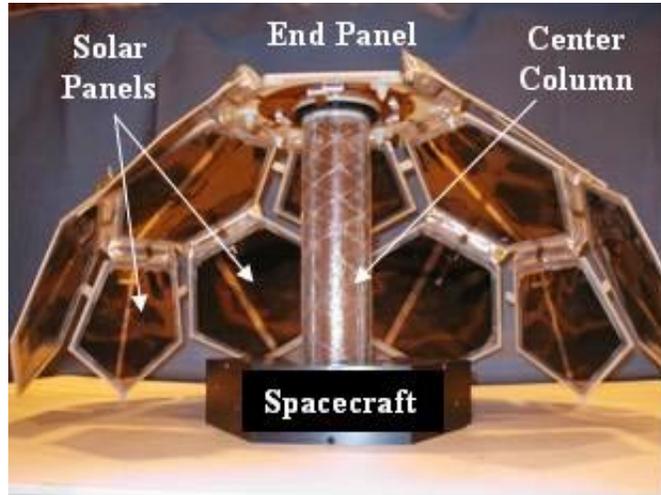
A prototypical center column test article was fabricated by ILC-Dover, Inc. of Dover, DE and provided to NASA Glenn Research Center (GRC) under contract NAS3-01115. Figure 3 shows a photograph of the center column unit in the NASA GRC film deposition chamber. The test article, measuring approximately 19 cm by 23 cm in its unrolled state, consists of  $2.54 \times 10^{-3}$  cm thick polyethylene terephthalate polyester (Mylar<sup>®</sup>) sheets sandwiching 0.051 cm diameter fiberglass ribs. To ease test article handling, the glass fibers were not impregnated with the UV-activated resin. The sheet edges are bonded using  $2.54 \times 10^{-3}$  cm thick Mylar<sup>®</sup> tape with pressure sensitive adhesive (PSA). On the sheet backside (inner side of rolled test article), there are two sets of 1.27 cm wide, power transfer flex circuit consisting of polyimide (Kapton<sup>®</sup>) encapsulated flat copper traces with a total thickness of 0.041 cm. The flex circuit is bonded to the back sheet using 2-side PSA tape. On the front side sheet (outer side of rolled test article), there are two sputter copper traces of widths 0.635 cm and 0.699 cm and thickness  $2.54 \times 10^{-4}$  cm. These traces run the length of the center column providing electrical bonding of the ITO-MgF<sub>2</sub> film. After depositing the ITO-MgF<sub>2</sub> film on the test article front side, the test article sheet was rolled and secured with Kapton<sup>®</sup> PSA tape to form a cylindrical column approximately 7.3 cm in diameter and 19 cm in length.

#### 2. Film Deposition & Characterization

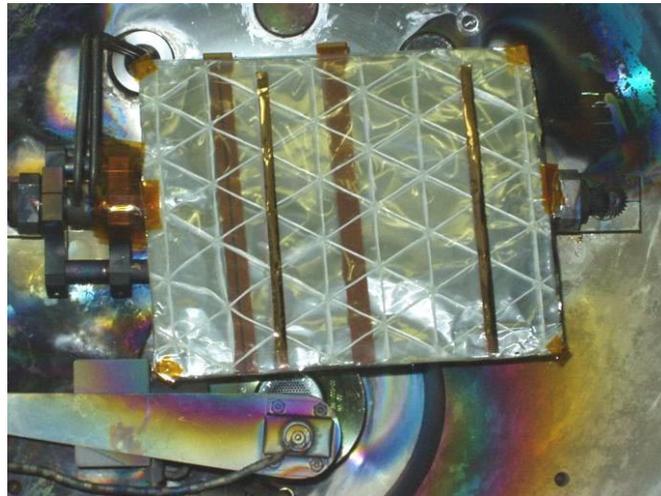
An Ion Tech Dual Beam Facility was used to deposit the film on the unrolled center column test article front side. This facility uses two ion sources to clean the target, clean the substrate for enhanced adhesion and to carry out the deposition without breaking vacuum. The facility was run on argon as the feed gas in a background of air for more complete oxidation. The pressure during the depositions was less than  $4.0 \times 10^{-4}$  Torr.

In order to obtain a mixed target, the individual deposition rates had to be determined for the ITO target and the MgF<sub>2</sub> target independently. Once the individual rates were determined, an aluminized piece of Kapton<sup>®</sup> was used to determine the beam center. Knowing the individual rates and the beam center, the 18 vol% MgF<sub>2</sub> required composition could be translated into angled wedges sitting on top of the ITO target. The mixed target was verified to give the proper deposition rate prior to coating the test article. The test article was then coated with a film of target thickness 500Å.

The film composition was based on calculations yielding 82 vol% ITO - 18 vol% MgF<sub>2</sub>. A quartz slide masked with Kapton<sup>®</sup> tape was used as a witness for thickness determination. The actual film thickness was determined using a "Dektak 6M Profilometer" by scanning from the tape protected region of the slide to the coated portion in four places. The measured film thickness was  $548\text{Å} \pm 68\text{Å}$ .



**Figure 2: PowerSphere Engineering Development Unit Hemisphere.**



**Figure 3: Center Column Test Article in Film Deposition Chamber.**

### 3. Fold Test Procedure

The rolled test article cylinder was flattened by hand to form a rectangular piece. The rectangular piece was folded by hand along its length to form one inward fold line and one outward fold line with respect to the ITO-MgF<sub>2</sub> coated outer sheet (see Figure 4). This “Z-folded” configuration is representative of center column stowed condition in a PowerSphere flight unit. During fabrication of a flight unit center column, the column core will be evacuated in vacuum chamber and then back filled with the relatively low vapor pressure inflatable material, i.e. heptane. During subsequent ground handling under ambient pressure, a compressive load of approximately  $1.01 \times 10^{+05}$  N/m<sup>2</sup> (atmospheric pressure) will be exerted on the Z-folded center column.

To simulate this stress loading on the center column, an aluminum fixture was built to uniformly support a 75 kg (734 N weight) static load on top of the 72.6 cm<sup>2</sup> center column Z-folded cross section (see Figure 5). The Z-folded test article was manually placed below the support fixture and the weights were carefully placed on top of the fixture. To avoid excessive test article ITO-MgF<sub>2</sub> film scratching akin to that reported in earlier testing<sup>6</sup>, several plastic sheets and a rubberized computer mouse pad were placed between the aluminum fixture and the laboratory table top. After a period of 5-minutes, the weights and fixture were carefully removed from the test article.

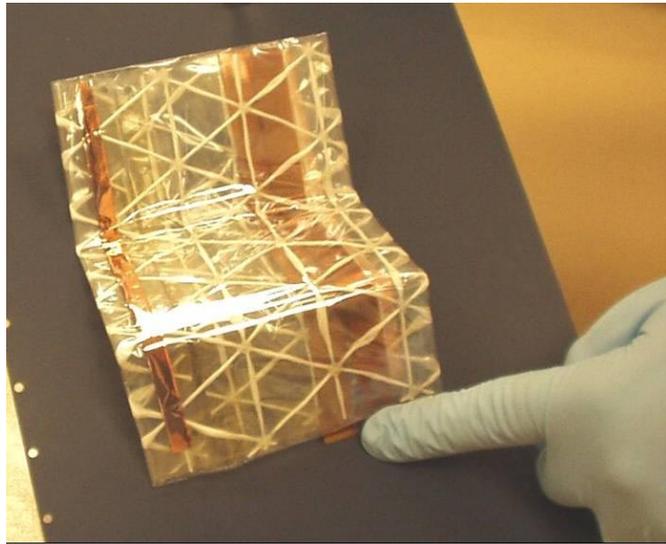
### 4. Vacuum Exposure Procedure

Approximately 4 months after completion of the fold test and subsequent optical and electrical inspections, the test article was exposed to high vacuum at room temperature for 75 days in a chamber pumped by an oil diffusion pump and liquid nitrogen trap. Chamber pressure, measured with an ionization gauge, was  $\sim 3.4 \times 10^{-07}$  Torr for most of this time; however, there were two excursions of at least several hours each to  $\sim 1 \times 10^{-05}$  Torr. Electrical inspections, as described in the following section, were made in ambient lab atmosphere before and after the vacuum exposure.

### 5. Film Inspection Techniques

The ITO-MgF<sub>2</sub> film was inspected in the region of fold lines using an “Olympus OM” stereo optical microscope operating at a magnification up to 102X. Photomicrographs were recorded using a “Canon E30” digital 35 mm camera.

ITO-MgF<sub>2</sub> film sheet resistance was measured under ambient laboratory lighting using a “Keithley 263” bi-polar DC source/calibrator operating in constant current calibrator mode. Electrical contact probes were placed at two points on the film. The probes were tipped with silicone rubber pads loaded with silver-coated copper powder. Guarded cabling between power supply and sample minimized errors due to cable leakage and capacitance. Current through the sample was determined from the setting of the source/calibrator, which is accurate to better than  $\pm 0.5\%$ . Voltage between contact probes was measured using a “Keithley 175” digital multimeter isolated by a unity-gain electrometer amplifier (built into the source/calibrator). This ensured that a negligible fraction of the current bypassed the sample. The amplifier/multimeter system is accurate to better than  $\pm 0.5\%$ , but electrostatic noise pickup introduced errors as large as  $\pm 2\%$ . Power supply polarity was switched to allow measurements with current flow in both directions. The film resistance, given by the measured voltage divided by the measured current, is



**Figure 4: Flattened and Z-folded Center Column Test Article.**



**Figure 5: Z-folded Center Column Test Article Under Static Load.**

reported as the average value of the bi-polar measurements. The root-sum-square difference in all bi-polar resistance values was 23%. This difference is ascribed to probe contact / film diode effects that can produce a few tenths of a volt difference in measured voltage. Figure 6 shows a photograph of the film resistance measurement apparatus while Figure 7 depicts the probe contact locations on the film with respect to test article fold lines and electrical bonding copper traces.

A potential error source is conduction through the polymer substrate. The authors attempted to measure the resistance between two points, ~6 cm apart, on the uncoated side of the sample and found it to exceed the upper limit of our equipment,  $\sim 1.5 \times 10^{13}$  ohms. The authors therefore estimate the error from substrate conduction to be <3% of our measured ITO-MgF<sub>2</sub> resistance values. The conductance of the electrical bonding copper traces was estimated from resistance measurements through the ITO-MgF<sub>2</sub> film between points directly above the copper traces. Measurements were made on the left-hand trace at points located immediately to the left of points A, B, and C on Figure 7 and also at equivalent points on the right-hand trace. The *largest* resistance obtained in these measurements was 5.5 ohms, showing that conductance through the copper exceeds that through the film by at least 6 orders of magnitude. Since these measurements were made after folding, the authors believe folding did not crack the copper traces.

### III. Film Sheet Resistivity Measurement and Active Control

#### A. Measurement Methods

Industrial production of transparent oxide coatings is typically done by sputtering from one or more magnetron sources rather than the ion beam sputtering method described in Section II.A.2. An important constraint on production methods is the high sensitivity of ITO-MgF<sub>2</sub>'s sheet resistivity to MgF<sub>2</sub>/ITO ratio<sup>9</sup>. To investigate ways to produce ITO-MgF<sub>2</sub> films with reproducible electrical properties, 2.5x2.5 cm<sup>2</sup> substrates were coated in a small vacuum chamber using two 5.1 cm diameter radio-frequency (13.56 megahertz) magnetron sputter sources. The sources, equipped with, respectively, ITO and MgF<sub>2</sub> targets, were driven by separate power supplies for adjustment of film composition. Deposition was carried out in argon gas at  $\sim 6 \times 10^{-3}$  Torr pressure. Most samples were deposited without addition of oxygen or air because this system produces highly transparent conductive ITO without it. Sample thickness was determined by readings of a single quartz crystal thickness monitor (QCM) located near the sample. The QCM had been calibrated separately for MgF<sub>2</sub> and ITO by measuring films of each, deposited on optically flat quartz, with a "Dektak IIA Profilometer"<sup>10</sup>. MgF<sub>2</sub>/ITO composition ratios were estimated from deposition rate measurements made on each gun at least once during each deposition run. (The authors had previously found the deposition rate to be approximately a linear function of radio frequency power).

Most of the small samples discussed in this report were approximately 400 Å thick, deposited on borosilicate glass. However, the authors have successfully coated quartz, Mylar®, Kynar®, and Upilex®. Thermocouple

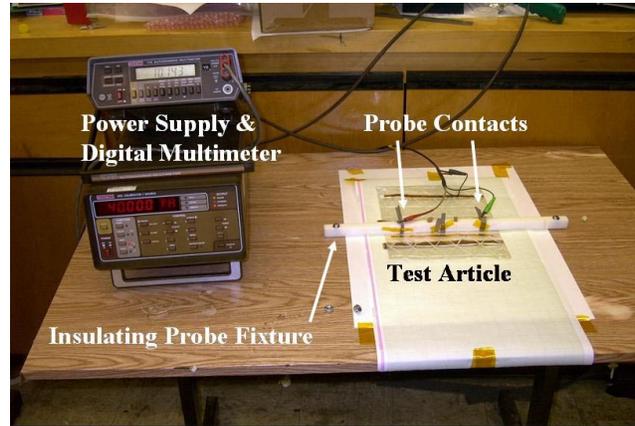


Figure 6: Film Resistance Measurement Apparatus.

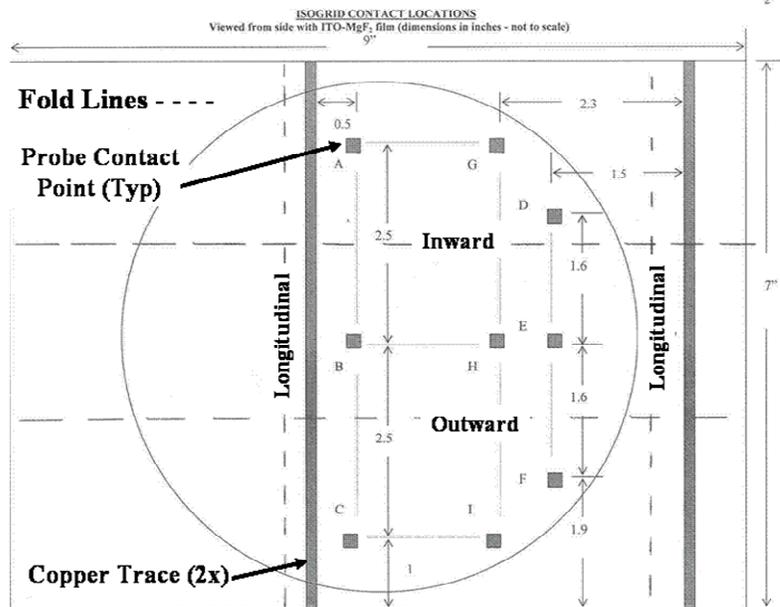


Figure 7: Film Resistance Measurement Locations (Dimensions in inches – 1 inch = 2.53 cm).

measurements indicate that substrate temperature is  $<40\text{ }^{\circ}\text{C}$  during deposition, so this method is suitable for coating polymers that cannot withstand high temperatures.

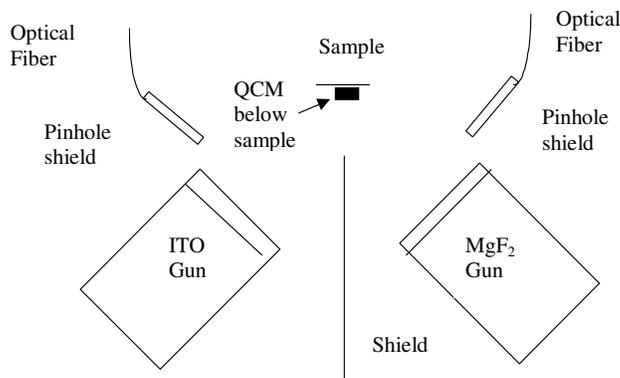
Our early tests showed that control of  $\text{MgF}_2/\text{ITO}$  ratio from QCM readings alone was not sufficient to obtain reproducible sheet resistivity. The authors therefore adapted a technique - plasma emission monitoring (PEM) - frequently used in the coating industry to aid production of transparent oxide films. This relies on the fact that the glowing plasma in front of a sputter target emits wavelengths characteristic of de-exciting atoms expelled from that target. Thus, the intensity of these emission lines is related to the sputtering rate of the target material. Light emitted by the plasma in front of each sputter source was collected by an optical fiber and fed to a two-channel “Ocean Optics S-2000” grating spectrometer located outside the vacuum chamber. The spectrometer was calibrated with known emission wavelengths from a mercury vapor lamp to an accuracy of  $\pm 0.5\text{ nm}$ . Experiments showed that each fiber received a negligible amount of light from the other sputter source. A schematic view of the deposition chamber and major components is shown in Figure 8.

Sheet resistivity measurements on the small samples were made at room temperature in ambient laboratory atmosphere and lighting conditions. Four-lead methods were used to eliminate the contribution due to resistance between contacts and the sample surface. During deposition, the substrate was covered with an aluminum mask to produce a sample measuring  $0.3 \times 1.9\text{ cm}^2$  with electrical contact arms along the edge. Spring-loaded pressure plungers tipped with conductive rubber pads made electrical contact to the samples. Direct currents of  $1 \times 10^{-12}$  to  $1 \times 10^{-4}$  amperes were provided by a “Keithley 263” bi-polar DC source/calibrator operating in constant current calibrator mode. Two “Keithley 614” high input resistance electrometers were used as unity-gain amplifiers to provide analog voltages at their low impedance preamp outputs that were essentially equal to the potentials of the two side contacts. The difference between these analog voltages was then measured with a “Keithley 175” digital multimeter. Guarded, shielded cabling minimized errors due to the high resistance of some samples. Voltage readings for both current directions were averaged to reduce errors from thermoelectric contact potentials and small electrochemically-generated currents in the sample. In most cases, the sheet resistivity of the substrate material was much higher than that of the film, so errors due to substrate conduction in parallel with the film were small. However, measurements on the reverse side of some coated glass substrates in humid weather showed sheet resistivity as low as  $\sim 1 \times 10^{+10}$  ohms/square. Thus, the error introduced by substrate conduction is  $\sim 1\%$  for reported values of  $1 \times 10^{+08}$  ohms/square (the main interest of our work) and  $\sim 10\%$  for reported values of  $1 \times 10^{+09}$  ohms/square. Our reported resistivity values  $> 1 \times 10^{+10}$  ohms/square may be seriously underestimated. While the instruments were accurate to better than  $\pm 0.5\%$ , some of our highest sheet resistivity values may have measurement errors as large as  $\pm 20\%$  due to excessive resistance at the film-contact boundary and finite size of the film’s contact arms.

## B. Active Control Methods

Stabilization of radio frequency power to the sputter sources proved inadequate to prepare films with reproducible sheet resistivity. Therefore the authors are investigating methods for active control of film properties based on measurements taken during deposition. Thus far the authors have tried regulation of the intensity of one spectral line each from the plasma discharges of the ITO and  $\text{MgF}_2$  sources. Lines chosen were those whose intensities showed the best correlation with sample sheet resistivity. Their wavelengths are  $453\text{ nm}$  from the ITO source and  $384\text{ nm}$  from the  $\text{MgF}_2$  source. The peak height of each line was reported by the spectrometer several times per second. The radio frequency power input to each sputter source was adjusted to keep the line intensity constant. Since the line intensities changed slowly over time, manual control was used in this feasibility study.

### PLAN VIEW



**Figure 8: Schematic Layout of Deposition Chamber (Width  $\sim 36\text{ cm}$ ).**

## IV. Results and Discussion

### A. Film Durability Testing Results

#### 1. Folded Polymer Substrate

For a baseline, several pre-fold photomicrographs were taken of the test article in the general vicinity of planned fold lines. Two of these photomicrographs, at optical magnification 24X and 63X, are shown in figures 9 and 10, respectively. Post-fold photomicrographs in the vicinity of inward and outward fold lines are shown in Figures 11 and 12, respectively. No damage to the ITO-MgF<sub>2</sub> film or Mylar® substrate is evident after folding based on this optical inspection.

Surface particular contamination can cause location perforation damage to the ITO-MgF<sub>2</sub> film and Mylar® substrate during Z-folding and Z-fold stack compression. An example of this perforation damage is shown in Figure 13. The dark region is a through-thickness hole while the light regions are torn Mylar edges that preferentially reflect light. During fabrication and handling of future flight hardware, clean room standards should minimize particulate contamination of PowerSphere polymer structures to minimize this type of film defect.

Film sheet resistance measurements were taken before and after test article folding. Results are provided in Table I (see Figure 7 for probe contact point locations). These data show that the film continuity is maintained with acceptable resistance level after test article folding. While no increase in film resistance was desired, the average increase in resistance over various film measurement locations was a factor of 14X. Since deposited film resistance can only be controlled to within an order-of-magnitude and film aging and light-exposure effects can change resistance values by an order-of-magnitude, a 10-100X increase in resistance due to folding is acceptable. An unusually high increase in film resistance (~300X) was seen between measurement locations A-B (adjacent to the copper trace). This reason for this behavior is unclear and is currently under investigation.

These results are much improved over those from earlier fold testing with a small polymer substrate coupon<sup>6</sup>. In the past testing, the ITO-MgF<sub>2</sub> coated Mylar® coupon was sharply folded under  $1.01 \times 10^{+05}$  N/m<sup>2</sup> loading. Post-fold measurements showed that the film was electrically insulating. In the current testing with a prototypical center column test article, several material layers are present. Under Z-folded compression, these layers allow for more graceful fold line film bend radii and hence, lower film stress level and a lower likelihood of film cracking<sup>11</sup>. In addition, the film was deposited over a copper trace sputtered on the Mylar® outer layer of the center column test article. It was speculated that the ductile copper trace would better handle folding stress and help to maintain electrical bonding across fold lines. However, the resistance data measured before and after folding do not indicate a clear benefit from the copper trace. Pre-fold film resistance values are about the same between points nearer and

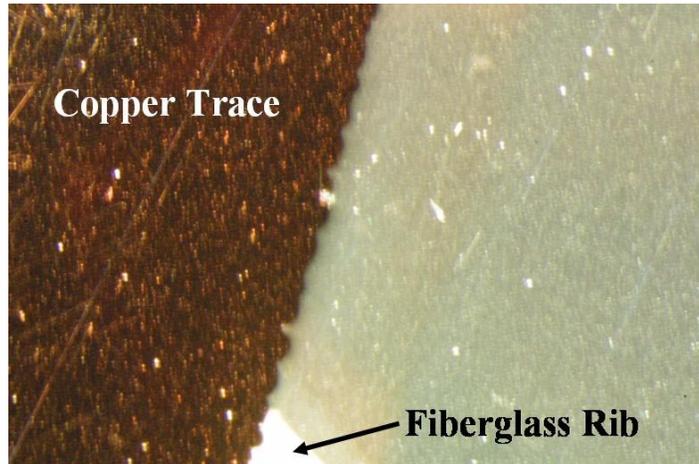


Figure 9: Pre-fold Photomicrograph at 12.5X Magnification.

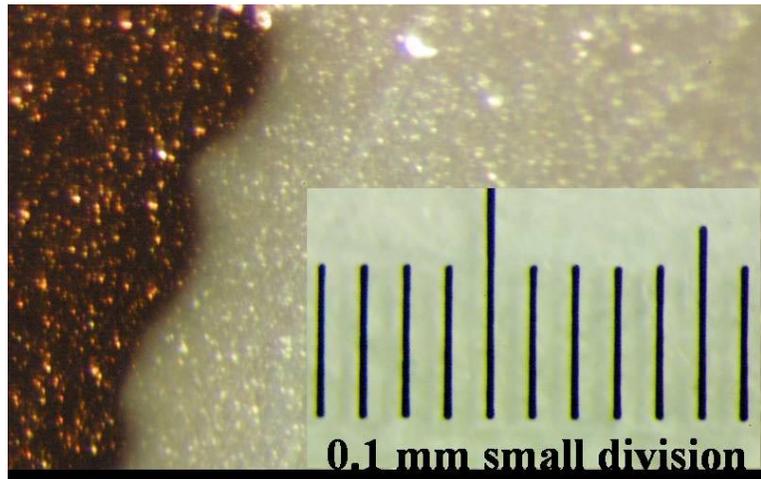


Figure 10: Pre-fold Photomicrograph at 31.6X Magnification (With Reticle).

farer from the copper traces, i.e. A-B and G-H. Also, the largest post-fold increase in film resistance (between A-B) occurred adjacent to the copper trace where one would expect to see a smaller increase.

## 2. Impact of Ambient and Vacuum Storage on Film Resistance

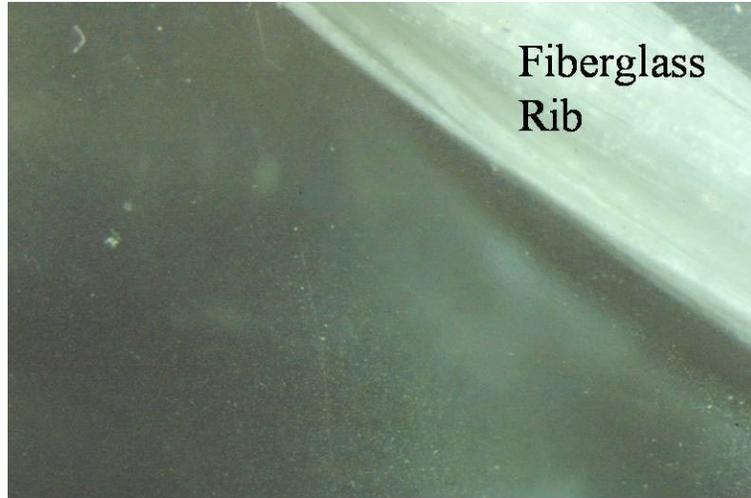
ITO-MgF<sub>2</sub> film sheet resistivity is known to increase over time under ambient conditions<sup>5</sup>. It is speculated that oxygen and water vapor diffuse into the film and the oxygen combines with free indium atoms. This reaction binds up two otherwise free valence electrons and hence, reduces the film electrical conductivity. To quantify this effect, film resistance measurements were repeated approximately 4-months following post-fold test measurements that were taken in late November 2003. Results show that on the average, the film resistance increased about 4x, over the various point to point measurement locations. Two measurements gave unusual results: that is, the film resistance from A-B actually decreased by 2X while that of B-C increased dramatically, i.e. 80X. The reason for these anomalous results is unclear and currently under investigation.

Film sheet resistance measurements made in ambient atmosphere before and ~20 minutes after the 75-day vacuum exposure are shown in Table II below (see Figure 7 for probe contact point locations). Surprisingly, these data show *increased* resistance after vacuum exposure rather than the decreased resistance after vacuum exposure reported previously<sup>5</sup>. The reason for this discrepancy is unknown. However, the authors note that all resistance measurements were made in ambient lab atmosphere rather than in vacuum and would not have detected a rapid change upon admission of air. In-situ resistance measurements inside a vacuum chamber are planned and will be reported at a later data.

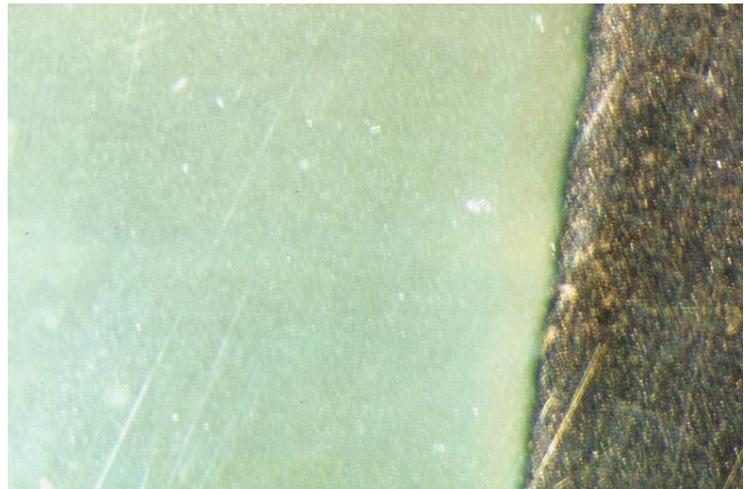
## B. Film Sheet Resistivity Measurement and Active Control Results

### 1. Measurement Methods

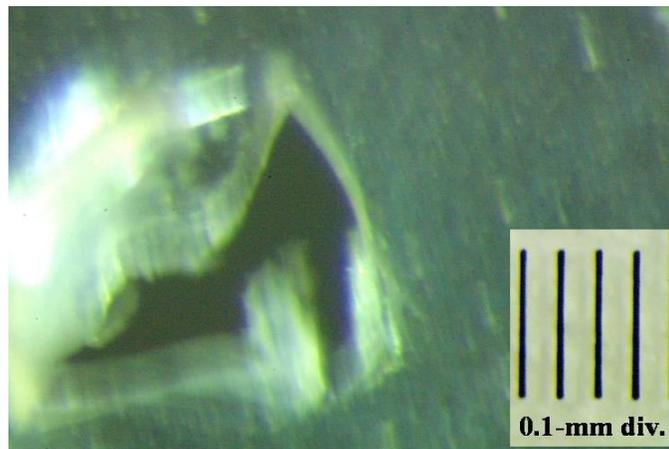
The intensity of the plasma emission lines chosen for monitoring is graphed vs wavelength in Figure 14, along with the wavelengths of relevant atomic emission



**Figure 11: Post-fold, Inward Fold Line Photomicrograph at 31.6X Magnification.**



**Figure 12: Post-Fold, Outer Fold Line Photomicrograph at 12.5X Magnification.**



**Figure 13: Post-Fold, Away From Fold Line Mylar® Substrate Damage at 31.6X Magnification.**

**Table I. Pre-Fold and Post-Fold Film Resistance Measurements**  
**ISOGRID WALL SAMPLE - RESISTANCE DATA**

Contacts	Before folding Nov. 22, 2003		After folding Nov. 26, 2003		Resistance After/Before	Contacts
	Current (A)	Resistance ( $\Omega$ )	Current (A)	Resistance ( $\Omega$ )		
A-C	1.00E-08	9.55E+07	5.00E-11	3.01E+10	320	A-C
A-C	1.00E-07	6.20E+07	5.00E-10	1.62E+10	260	A-C
A-B	1.00E-08	1.06E+08	5.00E-11	3.15E+10	300	A-B
A-B	1.00E-07	6.95E+07	5.00E-10	1.70E+10	240	A-B
B-C	1.00E-08	2.65E+07	1.00E-08	1.67E+08	6.3	B-C
B-C	1.00E-07	1.33E+07	1.00E-07	9.02E+07	6.8	B-C
D-F	1.00E-09	2.15E+08	1.00E-10	4.75E+09	22.1	D-F
D-F	1.00E-08	1.90E+08	1.00E-09	4.05E+09	21.4	D-F
D-E	1.00E-09	2.70E+08	5.00E-10	1.70E+09	6.3	D-E
D-E	1.00E-08	2.22E+08	5.00E-09	9.23E+08	4.2	D-E
E-F	1.00E-09	2.45E+08	5.00E-10	3.36E+09	13.7	E-F
E-F	1.00E-08	2.02E+08	5.00E-09	2.57E+09	12.7	E-F
G-I	1.00E-09	1.87E+08	5.00E-10	3.76E+09	20.1	G-I
G-I	1.00E-08	1.33E+08	5.00E-09	3.03E+09	22.9	G-I
G-H	1.00E-09	2.10E+08	1.00E-09	1.62E+09	7.7	G-H
G-H	1.00E-08	1.57E+08	1.00E-08	1.18E+09	7.5	G-H
H-I	1.00E-09	1.45E+08	5.00E-10	2.22E+09	15.3	H-I
H-I	1.00E-08	7.70E+07	5.00E-09	1.93E+09	25.1	H-I

**Table II. Film Resistance Measurements Before and After 75 Days of High Vacuum Exposure**

**ISOGRID WALL SAMPLE - RESISTANCE DATA**

Contacts	April 1, 2004		June 15, 2004 (10:35 AM)		Resistance After/Before	Contacts
	Current (A)	Resistance ( $\Omega$ )	Current (A)	Resistance ( $\Omega$ )		
A-C	1.00E-10	2.96E+10	1.00E-12	4.26E+12	144.03	A-C
A-B	1.00E-10	1.44E+10	1.00E-12	3.97E+12	276.31	A-B
B-C	1.00E-10	1.03E+10	1.00E-10	1.29E+10	1.26	B-C
B-C	1.00E-09	9.33E+09	1.00E-09	3.73E+09	0.40	B-C
D-F	1.00E-10	8.28E+09	1.00E-10	1.76E+10	2.13	D-F
D-F	1.00E-09	7.18E+09	1.00E-09	1.07E+10	1.49	D-F
D-E	1.00E-10	4.47E+09	1.00E-10	9.07E+09	2.03	D-E
D-E	1.00E-09	2.97E+09	1.00E-09	2.89E+09	0.97	D-E
E-F	1.00E-10	9.09E+09	1.00E-10	1.46E+10	1.61	E-F
E-F	1.00E-09	4.63E+09	1.00E-09	9.79E+09	2.11	E-F
G-I	1.00E-10	1.40E+10	1.00E-10	1.26E+10	0.90	G-I
G-I	1.00E-09	1.23E+10	1.00E-09	9.82E+09	0.80	G-I
G-H	1.00E-10	1.18E+10	1.00E-10	7.21E+09	0.61	G-H
G-H	1.00E-09	9.57E+09	1.00E-09	4.03E+09	0.42	G-H
H-I	1.00E-10	5.46E+09	1.00E-10	1.26E+10	2.30	H-I
H-I	1.00E-09	3.51E+09	1.00E-09	1.09E+10	3.11	H-I

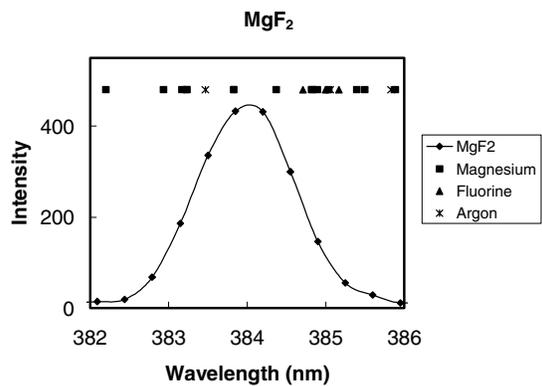
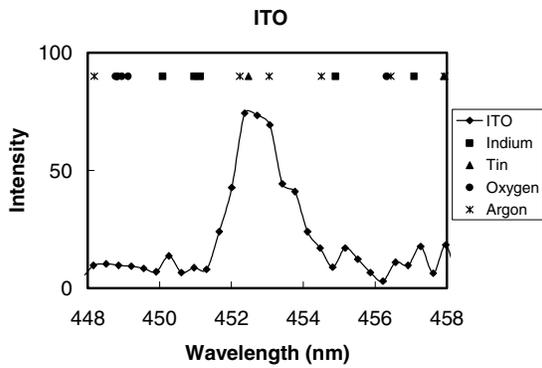


Figure 14: Spectra of Monitored Plasma Emissions (Intensity in Arbitrary Units).

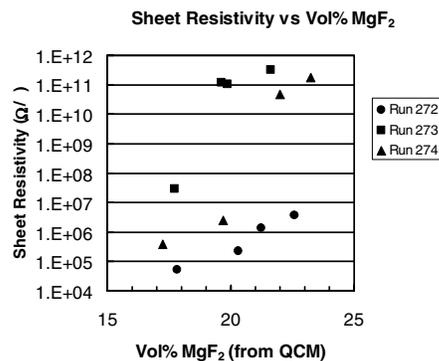
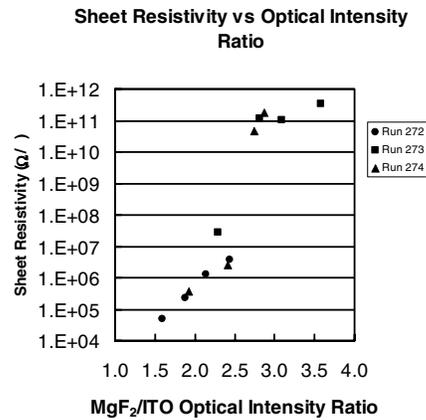


Figure 15: Sheet Resistivity vs  $MgF_2/ITO$  Intensity Ratio and Estimated  $MgF_2$  Concentration.

lines.<sup>12</sup> Based on these data, the authors believe the lines monitored are from tin and magnesium. In Figure 15 the authors show results of several deposition runs in which the  $MgF_2$  and ITO emission line intensities, rather than sputter source power inputs, were kept constant. Sheet resistivity of the resulting ITO- $MgF_2$  samples is plotted vs the ratio of  $MgF_2$  line intensity to ITO line intensity and vs the concentration of  $MgF_2$  deduced from QCM readings of each source's deposition rate. Note that sheet resistivity is more closely correlated to the line intensity ratio than to the deduced  $MgF_2$  concentration.

## 2. Active Control Methods

The data of Figure 15 suggest that coating reproducibility may be improved by regulation of plasma emission intensities, rather than power input to the sputter sources. However, the authors sometimes found large shifts of sheet resistivity relative to line intensity ratio and QCM data, usually after opening the vacuum chamber to remove samples. This is shown in Figure 16. In this case, sheet resistivity is poorly correlated with the plasma emission data as well as the QCM data.

Based on these results, it appears that plasma emission monitoring can facilitate production of ITO- $MgF_2$  coatings but probably must be supplemented by periodic in-situ measurements of the coatings' electrical or optical properties to "re-calibrate" the plasma emission monitor.

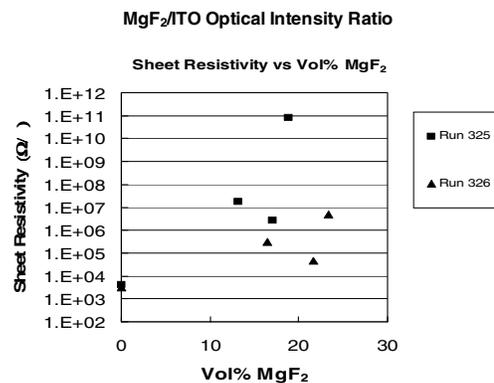
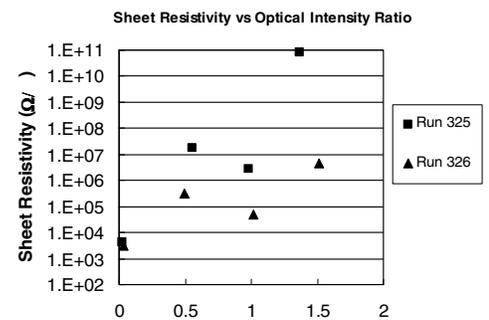


Figure 16: Data Similar to Figure 15, but Showing Poor Correlation of Sheet Resistivity with PEM and QCM Readings.

## V. Concluding Remarks

The durability of a 500Å thick, ITO-MgF<sub>2</sub> film on folded polymer substrates was investigated along with this film's resistance to vacuum exposure. Data collected indicate that acceptable film electrical bonding is maintained across polymer substrate fold lines to be encountered in the PowerSphere center column application. Film resistance changes after a 75-day high vacuum exposure were smaller than a factor of 3 except for two contact pairs that had shown an unusually large change when the sample was folded. However, the resistance measurements were made in ambient lab atmosphere; thus, further measurements with the sample in vacuum are needed.

Laboratory experiments showed that magnetron sputtering, a standard industrial coating technique, is promising for production of coated satellite parts provided problems with reproducibility of sheet resistivity can be solved.

Process control by plasma emission monitoring was investigated. Results showed that this method improves reproducibility but must be supplemented by actual in-situ measurement of one or more film properties, e.g. resistance and/or optical reflectance.

## VI. Future Work

A 500Å thick ITO-MgF<sub>2</sub> film has been deposited on prototypical PowerSphere solar cells with flex harness and included as part of the Materials International Space Station Experiment (MISSE-5) flight experiment to be launched to the International Space Station in the 2005 time frame. After exposure to the low Earth orbital environment for a year or more, the MISSE-5 samples will be returned to Earth to allow ITO-MgF<sub>2</sub> film characterization testing.

Additional ways to facilitate coating of satellite components will be investigated. Methods for measuring optical and electrical properties of ITO-MgF<sub>2</sub> in the deposition chamber will be tested for use in conjunction with plasma emission monitoring to improve reproducibility of coatings' electrical properties. A previously reported finding<sup>10</sup> that addition of air during deposition makes ITO-MgF<sub>2</sub>'s electrical properties less critically dependent on film composition will be followed up to see if this can simplify industrial production. Durability of films to vacuum ultraviolet light (VUV) exposure will also be tested.

## References

- <sup>1</sup>Simburger, Edward J., et al., "Development of a Multifunctional Inflatable Structure for the PowerSphere Concept," *3rd AIAA Gossamer Spacecraft Forum*, paper AIAA-2002-1707, Denver, CO, April 22-25, 2002.
- <sup>2</sup>Lin, John K., et al., "Development, Design and Testing of PowerSphere Multifunctional Ultraviolet-Rigidizable Inflatable Structures," *4th AIAA Gossamer Spacecraft Forum*, paper AIAA-2003-1707, Norfolk, VA, April 7-10, 2003.
- <sup>3</sup>Simburger, Edward J., et al., "Engineering Development Model Testing of the PowerSphere," *5th AIAA Gossamer Spacecraft Forum*, paper AIAA-2004-1570, Palm Springs, CA, April 19-22, 2004.
- <sup>4</sup>Ferguson, Dale F., internal communication, "Report on the Differential Charging of the PowerSphere With and Without Conductive Coatings," NASA Glenn Research Center, Photovoltaic & Space Environments Branch, Cleveland, Ohio, July 17, 2002.
- <sup>5</sup>Dever, Joyce A., et al., "Indium Tin Oxide-Magnesium Fluoride Co-Deposited Films for Spacecraft Applications," *International Conference on Metallurgical Coatings and Thin Films*, American Vacuum Society, San Diego, CA, April 24-26, 1996.
- <sup>6</sup>Kerslake, Thomas W., Waters, Deborah L., Scheiman, David A., and Hambourger, Paul D., "Durability of ITO-MgF<sub>2</sub> Films For Space-Inflatable Polymer Structures," paper AIAA-2003-5919, *1st International Energy Conversion Engineering Conference*, Portsmouth, VA, July 17 – August 21, 2003. (see also NASA/TM-2003-212512).
- <sup>7</sup>Uppala, Nicholas, Griffin, John, Vemulapalli, Jyothi, and Hambourger, Paul D., "Slightly Conductive Transparent Films for Space Applications- Manufacturability and Durability," *47<sup>th</sup> Annual Technical Conference Proceedings*, Society of Vacuum Coaters, 2004.
- <sup>8</sup>Griffin, John, Uppala, Nischala, Vemulapalli, Jyothi, and Hambourger, Paul D., "Transparent Arcproof Protective Coatings – Performance and Manufacturability Issues," ICPMSE-7, Protection of Materials and Structures from the Space Environment, Toronto, ON, Canada, May 10-13, 2004.
- <sup>9</sup>Cashman, T., et al., "Photoconductivity in Transparent Arcproof Coatings," *6th International Conference—Protection of Materials from Space Environment*, Toronto, Canada, May 1-3, 2002.
- <sup>10</sup>Cashman, Thomas, et al., "High Resistivity Transparent/Conductive Coatings for Space Applications," *Vacuum Technology and Coating*, September 2003, p. 38.
- <sup>11</sup>Banks, Bruce A., et al., "Ion Beam Sputter-Deposited Thin Film Coatings for Protection of Spacecraft Polymers in Low Earth Orbit," NASA TM-87051, January 1985.
- <sup>12</sup>NIST Atomic Spectra Database, [http://physics.nist.gov/cgi-bin/AtData/lines\\_form?XXR0q0qF](http://physics.nist.gov/cgi-bin/AtData/lines_form?XXR0q0qF)

# REPORT DOCUMENTATION PAGE

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<b>13. ABSTRACT</b> ( <i>Maximum 200 words</i> )  Multi-kilogram class microsattellites with a PowerSphere electric power system are attractive for fulfilling a variety of potential NASA missions. However, PowerSphere polymer surfaces must be coated with a film that has suitable electrical sheet resistivity for electrostatic discharge control, be resistant to atomic oxygen attack, be transparent to ultraviolet light for composite structure curing and resist ultraviolet light induced darkening for efficient photovoltaic cell operation. In addition, the film must be tolerant of polymer layer folding associated with launch stowage of PowerSphere inflatable structures. An excellent film material candidate to meet these requirements is co-sputtered, indium oxide (In <sub>2</sub> O <sub>3</sub> ) - tin oxide (SnO <sub>2</sub> ), known as "ITO", and magnesium fluoride (MgF <sub>2</sub> ). While basic ITO-MgF <sub>2</sub> film properties have been the subject of research over the last decade, further research is required in the areas of film durability for space-inflatable applications and precise film property control for large scale commercial production. In this paper, the authors present film durability results for a folded polymer substrate and film resistance to vacuum UV darkening. The authors discuss methods and results in the area of film sheet resistivity measurement and active control, particularly dual-channel, plasma emission line measurement of ITO and MgF <sub>2</sub> plasma sources. ITO-MgF <sub>2</sub> film polymer coupon preparation is described as well as film deposition equipment, procedures and film characterization. Durability testing methods are also described. The pre- and post-test condition of the films is assessed microscopically and electrically. Results show that a ~500Å ITO-18vol% MgF <sub>2</sub> film is a promising candidate to protect PowerSphere polymer surfaces for Earth orbit missions. Preliminary data also indicate that in situ film measurement methods are promising for active film resistivity control in future large scale production. Future film research plans are also discussed.			
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